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diamond-to-diamond contact

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(54) A METHOD OF PREPARING A DIAMOND-METAL COMPACT

(71) We, INSTITUT FIZIKI VYSOKIKH DAVLENY AKADEMII NAUK SSSR., of Moskovskaya oblast, Podolsky raion, Akademgorodok, Union of Soviet Socialist Republics (U.S.S.R.), a State enterprise organised and existing under the laws of the U.S.S.R., do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The present invention relates to a method of preparing a diamond-metal compact. Such compacts comprise a composite of diamond crystals and a metallic binder which is dispersed mainly in the interstices between individual diamond crystals to bind the crystals together.

Due to a high concentration of diamonds, these compacts resemble single-crystal diamonds in such properties as abrasion resistance, hardess and elastic modulus. The extent of the resemblance depends substantially on the process used to form the compacts.

Applications of compacts are similar to those of large single crystals of diamond, i.e. they are useful for processing hard and brittle materials such as glass and ceramics, for processing or drilling rocks, and for manufacturing articles which require to possess extreme hardness, high abrasion resistance and compression strength, as, for instance, movable parts of precision intruments and most critical parts of high-pressure vessels. These compacts are much more easily available and less expensive than large single-crystal diamonds, since the diamond powder necessary for their production is now produced in large quantities.

There have previously been proposed various processes for producing diamond-metal compacts by impregnating diamond powder with a metallic binder; these processes provide for simultaneous application of a high pressure and temperature to a system comprising diamond powder and a metallic binder. In such a system there is used a mechanical mixture of diamond powder and metal powder, i.e. the metallic binder, in this case, is incorporated into the diamond powder prior to the subjection of temperature and pressure. The content of the metal powder is less than or equal to 50% by volume, e.g. 20 to 30% by volume. The mixture thus obtained is first subjected to a pressure ranging from 70 to 85 kbar and then heated, still under said pressure, to a temperature ensuring melting of the metal so that the molten metal fills the inter-grain diamond pores.

These prior-art processes have a disadvantage which resides in an insufficient packing density of the diamond grains in the final compact and occurrence of metallic interlayers in the regions of diamond-to-diamond contact, whereby the hardness and other mechanical characteristics of the compact are impaired. In fact, it is not possible to attain the maximum packing density of diamond grains simply by the application of pressure to the initial diamond-metal mixture since it is hindered by the metallic powder present in a considerable amount in the mixture.

When the mixture is heated under pressure, the metal powder melts and fills the pores between the diamond grains, but part of the metal still remains in the form of a film in the regions where diamond grains contact one another.

The diamond grains are thus separated from one another by this metal film, whereby the hardness of the resulting compact is lowered, as well as its compression strength and elastic modulus, since these properties of the metal are substantially less than those of the diamond particles.

Another disadvantage of the prior-art processes for the production of compacts from a diamond-metal powder mixture resides in that during melting of the metal powder, which is intimately dispersed throughout the

elastic modulus = applied stress / Δ in shape

diamond-metal mixture, absorbed gases, foreign atoms, and other contaminants present on the surface of the diamond particles cannot find their way out of the mass and remain trapped within the compact. This results in microporosity which leads to decreased hardness, compression strength, and elastic modulus.

It is an object of the present invention to obviate or mitigate the above disadvantages, and to provide a compact having high abrasion resistance and improved hardess, compression strength and elastic modulus.

It has previously been proposed to reconstitute diamond powders by placing a mass of finely divided diamonds adjacent a mass of a metallic solvent for the diamond and subjecting the masses to high temperature and pressure so as to dissolve the diamond in the metallic solvent. Upon cooling, the diamond recrystallises from the solvent so that new particles of diamond of larger size than those initially present are produced.

According to the present invention there is provided a method of preparing a diamond-metal compact comprising placing a mass of particulate diamond and a mass of a metallic binder material as hereinafter defined adjacent one another, and subjecting the masses to a pressure greater than 10 Kb at a temperature sufficient to melt the binder material so that on cooling the diamond particles bed in the binder material.

The mass of metallic binder material may be disposed in the system in various ways, e.g. under a layer of diamond powder, above a layer of diamond powder, or along the side face of a diamond briquette so that the metallic binder material is in contact with the diamond briquette.

By the term "metallic binder" as used in this specification and in the appended claims is meant a metal or alloy having a boundary angle of wetting less than 90° in respect of diamond. Examples of such metals are nickel, cobalt, iron, manganese and chromium. As the metallic binder material use may be made of an alloy of a diamond-wetting metal or an alloy of a diamond-wetting metal with a non-diamond-wetting metal or with a non-metal. Examples of such alloys are nickel-chromium, nickel-manganese, nickel-copper, cobalt-copper, titanium-copper, zirconium-copper, nickel-copper-aluminium and nickel-copper-silicon. The wetting metal content in said alloys may be varied, but it is desirable that it be of at least 15% by volume, and preferably between 15% and 30% by volume. It is possible to employ an alloy having a wetting metal content less than 15%, but in that case the resulting properties will be less pronounced.

The amount of metallic binder is preferably selected so that it is sufficient to fill all the voids between the diamond particles. It has previously been shown that voids in diamond powder compressed under a pressure of at least 10 kbar may amount to 10 to 30% by volume depending on the grain size of the diamond. Therefore, the volume of the metallic binder material may constitute 10 to 30% of the volume occupied by the diamond powder when the latter has been compressed. If the binder material is employed in an amount greater than 30%, then a layer of, for example, boron carbide, B_4C, in solid particulate form may be disposed on an opposite side of a diamond-powder briquette from the binder material. This layer will take up excess binder material after it has melted.

The starting diamond particle size is selected depending on the intended application of the diamond-metal compact to be produced. If it is intended for use as an abrasive, a large diamond particle size is preferred, e.g. 15 to 250μ. If the material is required to possess a high compression strength and hardness, it is advisable to select a finely grained diamond powder having a particle size of 0.5 to 15μ. In many applications, it is advantageous to employ a mixture of diamond powders of various particle sizes which makes it possible to increase the proposition of the diamond in the compact to a maximum since, in doing so, voids between coarse diamond particles become filled with fine diamond particles. A preferential ratio of large and fine particles ranges from 3:1 to 10:1 Volumetric proportion of large and fine grains is 70—80 and 30—20% respectively.

Met to use two particle sizes

The pressure to which the masses of particulate diamond and metallic binder material are subjected should be at least 10 kbar and is usually selected within the range from 10 to 50 kbar. Pressures above 50 kbar facilitate packing of the diamond particles, especially fine ones, and the impregnation thereof with the metallic binder material. However, it should be understood that, though under pressure of 50—100 kbar and over, better packing of diamond powder is obtained and the final product is of higher quality, the service life of the chamber wherein the process is performed is substantially reduced.

Pressure selection is also influenced by the diamond particle size. The smaller the particles, the higher the pressure applied and, vice versa, the larger the particles, the lower the pressure, since large particles are more susceptible to mechanical damage and cracking than small particles, when compressed in a pressure chamber. In practice, when diamond is used having a particle size of from 0.5 to 15μ, the pressure employed may be from 30 to 50 kbar, whereas for diamond having a particle size of from 15 to 250μ, the pressure employed may be from 10 to 20 kbar. When a mixture of diamond particles of various sizes is used, the recommended pressure range may

Particle size vs pressure

be substantially exceeded. Properly selected pressures ensure the production of high quality compacts and, at the same time, enable the service life of a pressure chamber to be extended.

The present invention makes it possible, depending on the particle size of the diamond and on the binder employed, to produce a diamond-metal compact possessing extremely high mechanical properties: hardness of 95—96 units HRA (for diamond grains of 3 to 5μ size; HRA—Rockwell hardness, scale A), compression strength of 450 to 550 kg/mm^2, and elastic modulus of 55,000 kg/mm^2 (for diamond grains of 10 to 15μ size). When a cone made of a diamond-metal compact (with a diamond particle size 3 to 5μ) is pressed in a tungsten-carbide alloy of the composition 94% WC and 6% Co, said diamond-metal compact can withstand a contact pressure of up to 10,000 kg/mm^2.

In addition to high mechanical characteristics, the diamond-metal compacts produced by the method of the present invention posses satisfactory resistance to the effect of shock loads.

An important feature of the present method of producing a diamond-metal compact resides in a possibility of manufacturing a compact having a volume of 10 cm^3 or more. The volume of a compact is defined by the volume of a pressure chamber used to form it and, since the pressures required in the present method can be relatively small, the dimensions of the pressure chamber may be easily increased to produce a volume of 10 ccm or more.

The excellent mechanical properties of the diamond-metal compacts produced by the present method result from the fact that the initial masses of particulate diamond and metallic binder material are not mixed prior to the application of heat and pressure. The two masses are simply in contact with each other, and the penetration of the binder material into the mass of particulate diamond is effected under a pressure of at least 10 kbar and at a temperature sufficient to melt the metallic binder material.

Therefore, the diamond powder is closely packed by the pressure applied thereto prior to its penetration by the metallic binder material. This embodiment makes it possible to obtain the densest packing of diamond grains and form a solid diamond substrate with a plurality of diamond-to-diamond contact sites. The binder material is incorporated into the packed diamond mass, which is still under pressure, and the molten binder material fills spaces between the diamond particles. The diamond substrate formed under pressure is bonded by the solidified metallic binder material so that the diamond particles become bedded therein. Diamond-metal compacts of this type function rather well under compression loads, since the loads are taken up mainly by the diamond substrate, the diamond-to-diamond contact regions of the latter not being weakened by metallic interlayers. The metallic binder material only secures individual particles of the diamond, and prevents them from mutually displacing.

Furthermore, during the impregnation of the particulate diamond mass with the metallic binder material, superficial contaminants of the diamond particles and other foreign matter pass into the metal melt, becoming concentrated at the melt front. The contaminants and other foreign matter are thus carried outside the mass of diamond.

Owing to this phenomenon, the major part of the diamond-metal compact now contains few impurities. The surface of the diamond-metal compact, i.e. that portion where the melt front stopped and where contaminations and defects are concentrated, may be relativel easily removed by subsequent mechanical processing of the compact.

In order to reduce the consumption of diamond, the particulate diamond can be replaced, at the portion of the compact at a purported site of the melt stoppage, where the melt point is expected to stop, by boron carbide, B_4C, powder. In this way, the contaminated layer to be removed will be constituted by the much less expensive, boron carbide, and diamond wastage is reduced.

Therefore, the method of the present invention enables the production of a diamond-metal compact with a predetermined internal structure: a solid diamond substrate with a plurality of direct diamond-to-diamond contacts, and a metallic binder material distributed within the pores of the diamond substrate.

The following is the description of an exemplary embodiment of the method of the present invention, with reference to the drawing, wherein a pressure chamber is shown.

The starting components of a diamond-metal compact, viz. a mass of diamond powder 1 of a selected particle size or a mixture of diamond powders of various particle sizes, and a mass of a metallic binder material 2, are placed in a graphite container-heater 3. The metallic binder material has the form of pre-pressed powder or chips molded to a predetermined shape and dimensions during the pressing, or in the form of a monolithic piece of metal shaped to a predetermined configuration and dimensions by turning. The mass of the metallic binder material is placed within the graphite heater so as to contact the mass of diamond powder. From the opposite side, with respect to the binder material, a layer 4 of boron carbide, B_4C, is placed in contact with the mass of diamond powder. The graphite heater walls may be electrically insulated from the heater contents by means of gaskets 5 made of mica or hexagonal boron

nitride. To prevent the diamond-metal compact from absorbing gases evolved from the graphite heater 3 during heating, the heater 3 is isolated mechanically from the contents by means of a foil 6 made of a refractory metal such as tantalum or tungsten. The filled heater 3 is then covered with a graphite lid 7.

The heater 3 with its contents is then placed in a container 8 made of katlinite or pyrophillite. Katlinite or pyrophillite are employed for transmitting and equalizing pressure applied to the reaction space. The heating is effected by passing an electric current through the graphite wall along the length of the heater 3.

The method of producing a diamond-metal compact by the present embodiment consists in the following steps performed in this order: the container 8 is first subjected to a pressure of at least 10 kbar. The diamond powder is thus closely packed. The heater 3 is then heated to a temperature ensuring melting of the metallic binder material 2. The molten binder material 2 impregnates under pressure the packed diamond powder 1. The system is maintained at the impregnation temperature generally for a period of from 30 sec. to 3 min. Then the heating is discontinued and, after solidfying the metallic binder material for an additional 0.5—2 min, the pressure is brought to zero.

It should be noted that the present method also makes it possible to produce a diamond-metal compact of a predetermined shape. A required shape may be obtained by means of ceramic inserts placed into the graphite heater prior to charging with the diamond. A similar result can be obtained by suitably shaping the inner cavity of the graphite heater which may thus be made, for example, spherical or octahedral, depending on the required shape of a diamond-metal compact.

If it is desired to produce a shaped diamond-metal compact with an internal hole or depression, the mass of metallic binder material may be molded into the shape of such a hole or depression and placed within a recess in the mass of diamond powder. To produce a diamond-metal compact having a relief surface, a corresponding relief may be made on the surface of the mass of binder material contacting the mass of diamond powder. Such relief is reproduced on the surface of a diamond-metal compact manufactured by the method of the present invention.

Embodiments of the present invention will now be described by way of illustration in the following Examples.

Example 1

A diamond-metal compact is produced in the apparatus shown in the accompanying drawing.

The internal cavity of the graphite heater 3 has a diameter of 30 mm and a height of 30 mm. The starting materials are diamond powder 1 having a particle size of 150μ and metallic binder material 2, viz. an alloy containing zirconium and 50% by weight of copper. The filled heater 3 is subjected to 10 kbar pressure at a temperature of 1,250°C. The system is maintained at this temperature for 1.5 min. The resulting material is employed for making tools adapted for processing stone.

Example 2

The method is performed as in Example 1. The starting materials are: diamond powder having a particle size of 150μ; metallic binder material comprising metallic nickel. Process conditions: pressure 20 kbar; temperature 1,500°C; treatment time 1.5 min. The resulting material has high abrasion properties and a high resistance to shock loads.

Example 3

The internal cavity of the graphite heater 3 has a diameter of 15 mm and a height of 15 mm. Diamond powder having a particle size of 10 to 15μ and a metallic binder material comprising a titanium alloy with 40% by weight of copper are subjected to 30 kbar pressure and 1,200°C temperature. Treatment at this temperature takes 1 min. The material thus obtained has a compression strength of 500 kg/mm^2 and an elastic modulus of 55,00 kg/mm^2.

Example 4

The reaction cavity has the same dimensions as in Example 3. Diamond powder having a particle size of 3 to 5μ and a binder material comprising a titanium alloy with 45% by weight of copper are subjected to 50 kbar pressure and 1,150°C temperature for 1 min. The resulting material has an HRA hardness of 96.

WHAT WE CLAIM IS:—

1. A method of preparing a diamond-metal compact comprising placing a mass of particulate diamond and a mass of a metallic binder material as hereinbefore defined adjacent one another, and subjecting the masses to a pressure greater than 10 Kb at a temperature sufficient to melt the binder material so that on cooling the diamond particles bed in the binder material.

2. A method as claimed in claim 1, wherein the masses are subjected to a pressure in the range from 10 to 50 Kb.

3. A method as claimed in claim 2, wherein the pressure is 30 Kb.

4. A process as claimed in any preceding claim, wherein the binder material is metallic nickel, metallic cobalt or metallic manganese.

5. A method as claimed in any one of

claims 1 to 3, wherein the binder material comprises an alloy having a boundary angle of wetting less than 90°.

6. A method as claimed in claim 5, wherein the binder material is a zirconium alloy containing 50% by weight of copper.

7. A process as claimed in claim 5, wherein the binder material is a titanium alloy containing 40 to 45% by weight of copper.

8. A method as claimed in any one of the preceding claims, wherein the particulate diamond has a particle size in the range from 0.5 to 250μ.

9. A method of preparing a diamond-metal compact substantially as hereinbefore described with reference to any one of the Examples.

10. A diamond-metal compact whenever produced by the method claimed in any one of the preceding claims.

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1382080 COMPLETE SPECIFICATION

1 SHEET *This drawing is a reproduction of the Original on a reduced scale*